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RECENTLY PUBLISHED RESEARCH OF THE MENDELEYEV INSTITUTE OF CHEMICAL TECHNOLOGY, MOSCOW, USSR, PART II

"Absorption of Sound in Binary Gaseous Mixtures," B. P. Kundryavtsev, Lab Phys Chem, D. I. Mendeleyev Chem Tech Inst, Moscow

"Zhur Eksper Teor Fiz" Vol 17, 1947, pp 294-300

Measurements were made by the acoustic interferometer method according to Belyavskaya (1936) by using a vibrating quartz source and a reflector with a micrometric displacement accurate to 0.005 mm, or by the method of Pielemeier (1929) and Pielemeier, Saxton and Telfair, at a frequency of 947 kc. In the system $N_2 + H_2$, at 17°, the absorption coefficient α is a linear function of the composition, within the limits of experimental error. In air $+ CO_2$, at 20°, the curve of α is of the parabolic type, concave to the axis of composition, in contradiction to the findings of Rogers (1934) which are subject to caution because of the inadequacy of the radiometer method. In $H_2 + CO_2$ α passes through a minimum at about 70-80% H_2. Since collisions between H_2 and CO_2 are more effective in excitation of vibrational states of CO_2 than collisions between CO_2 and CO_2, it can be assumed that addition of H_2 to CO_2 shortens the time of establishment of equilibrium distributions of energy and thus suppresses the corresponding losses of acoustic energy; this causes α to decrease with increasing amount of H_2, until complete disappearance of the absorption due to real relaxation of equilibrium distribution. On further addition of H_2, the normal additivity of α reappears. This viewpoint is illustrated by super-

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position of the $H_2 + N_2$ and $H_2 + CO_2$ plots, showing practical coincidence with the $H_2 + N_2$ curves of the branch beyond the minimum which marks the complete annulment of the dispersion effect. The anomalous absorption of sound in H_2 cannot be due to losses related to a lag of distribution of energy between the degrees of freedom of that molecule.

"Chemical Compounds of Benzene With Halogen Derivatives of Methane," A. F. Kapustinskiy, S. I. Drakin, D. I. Mendeleyev Chem Tech Inst, Moscow

"Bull Acad Sci URSS, Classe Sci Chim" 1947, pp 435-42

Melting diagrams were constructed by freezing in liquid air and thermal analysis in spontaneous warming up; this technique, eliminating under cooling is more reliable than observations of beginning freezing on cooling; the temperature is accurate within 0.1°, composition within 1%. Compounds investigated were C_6H_6-CCl_4, C_6H_6-CBr_4, C_6H_6-$CHCl_3$, C_6H_6-CH_2Cl_2, and C_6H_6-CI_4.

"Preparation of Silicic Acid Esters of Synthetic Hydroxy Acids Obtained by Air Oxidation of Paraffin," A. P. Kreshkov, D. I. Mendeleyev Chem Tech Inst, Moscow

"Zhur Obshch Khim" Vol 17, 1947, pp 81-6

$SiCl_4$ and $Si(OEt)_4$ were made to react with pilot-plant products (hydroxy carboxylic acids and their Me esters) obtained by oxidation of paraffin at 160° and corresponding in properties to materials used in earlier studies. $SiCl_4$ failed to yield the expected esters; its action was that of initial dehydration followed by addition of $SiCl_4$ to the double bond as Cl-$SiCl_3$, the latter being converted to -CHCl-$CH(SiO_2H)$- after treatment with H_2O. $(EtO)_4$ Si reacts with the HO acids with formation of free silicic acid and of a viscous product which is apparently a mixture of condensates of partially hydrolyzed silicic esters. Only the reaction of $(EtO)_4Si$ with Me esters of the OH acids led to the desired silicic esters.

"Polymerization of Methyl Vinyl Ether," I. P. Losev, E. B. Trostyanskaya, D. I. Mendeleyev Chem Tech Inst, Moscow

"Zhur Obshch Khim" Vol 17, 1947, pp 122-9

No polymerization was observed in the presence of Bz_2O_2, urea peroxide, or H_2O_2, or on heating 45 days at 80°, or in the presence of 1% $ZnCl_2$ or $CuCl_2$. With $AlCl_3$, 0.01 and 0.02%, the yields, Y, of polymer in 16 hours were 72.7 and 75.7%, respectively. With $FeCl_3$, 0.01 and 0.02%, 12 and 16 hours, respectively, Y = 73.4 and 74.9, respectively. With PCl_3, a sirupy dark brown polymer is formed. The most effective catalyst is $SnCl_4$; with 0.12%, Y = 94.0%. Copolymerization in the

presence of 1% Br_2O_2, with CH_2:$CMeCO_2Me$ and with allyl methacrylate gave varying yields of copolymers, none of which was soluble in organic solvents. Oxidation of the methyl vinyl ether polymer with 30% H_2O_2 gave no dibasic acid but did produce HCO_2H and AcOH; this confirms Standinger's oxidation scheme and the "head-to-tail" structure of the polymer. In an attempt to reproduce the polymer by methylation of the polymer of vinyl alcohol, the latter was prepared by saponification of high-molecular polyvinyl acetate in an N atmosphere and subjected to several consecutive non-destructive methylations with Me_2SO_4 in slightly alkaline medium.

"Preparation of Oxides of Tertiary Aromatic Amines: Methyldiphenylamine Oxide," V. N. Belov, K. K. Savich, D. I. Mendeleyev Chem Tech Inst, Moscow

"Zhur Obshch Khim" Vol 17, 1947, pp 257-61

Although $PhNMe_2$ is rapidly oxidized by 3% H_2O_2, this is not true of Ph_2NMe, which is essentially unchanged after 20 hours heating with dilute H_2O_2. When perhydrol (presumably 30% H_2O_2) and Ac_2O were shaken with Ph_2NMe, however, a vigorous reaction took place, which was moderated by cooling to 40-50°; after standing 1-2 hours, the somewhat less colored solution was treated, with efficient cooling, with aqueous KOH and KOAc was separated; the residual solution was extracted with $CHCl_3$ repeatedly; the dried extract, evaporated in vacuo, gave 88% Ph_2MeNO needles. The air-dried product is a hydrate.

"Addition Products of Phenylmagnesium Bromide and Oxides of Dimethylaniline and Methyldiphenylamine," V. N. Belov, K. K. Savich, D. I. Mendeleyev Chem Tech Inst, Moscow

"Zhur Obshch Khim" Vol 17, 1947, pp 262-8

The products give on hydrolysis a quantitative recovery of the original oxides, benzene, and basic Mg salts. Heating the adduct in dry solvents resulted, in the case of the Me_2NPh product, in recovery of PhCH and $PhNMe_2$ in good yields. The adducts may be considered as ammonium-type salts, with a positive charged complex ion and negative Br ion, i.e., $(PhMe_2NCHPh)Br$.

"Anthraquinones Series: I, 1,4-Anthraquinonedisulfonic Acid," V. V. Kozlov, D. I. Mendeleyev Chem Tech Inst, Moscow

"Zhur Obshch Khim" Vol 17, 1947, pp 289-98

1,4-Anthraquinonedisulfonic acid was most conveniently prepared by the reaction of 1,4-dichloroanthraquinone with Na_2SO_3. Describes reaction procedure.

"Mechanism of the Friedel-Crafts Reaction: V, Complex Compounds of Benzene and Toluene With Aluminum Bromide," V. V. Korshak, N. N. Lebedev, S. D. Fedoseyev, D. I. Mendeleyev Chem Tech Inst, Moscow

"Zhur Obshch Khim" Vol 17, 1947, pp 575-83

Judging by the strict additivity of the molecular reactions, solutions of $AlBr_3$ in benzene or PhMe do not contain truly definite compounds but represent an equilibrium of the type $Al_2Br_6 + nC_6H_6 \rightleftarrows Al_2Br_6(C_6H_6)_n$ shifted substantially to the left-hand side. When such a solution is treated with HBr, however, profound changes occur, leading to a complex which separates as an oil, characterized by electroconductivity and having the composition $Al_2Br_6.Ar_6.HBr$; the overall system is best considered an equilibrium similar to that given. Describes preparation of $AlBr_3$ and $AlCl_3$ and the reaction processes.

"Sulfonating Action of Dialkyl Sulfates: IV, Reaction of Dimethyl and Diethyl Sulfates With Para-Thiocresol and 2-Thionaphthol," V. N. Belov, M. Z. Finkel'shteyn, D. I. Mendeleyev Chem Tech Inst, Moscow

"Zhur Obshch Khim" Vol 17, 1947, pp 741-6

Contrary to phenols, their thio analogs react with R_2SO_4 in the same manner regardless of whether or not alkali is present: The thiophenols are alkylated and the products add R_2SO_4. The difference is only quantitative: In the absence of alkali the latter reaction is the more rapid one, while the reverse is true in the presence of alkali. Gives reaction processes and properties of derivatives.

"Anthraquinone Series: V, ms-Sulfonic Acid of Anthrone," V. V. Kozlov, D. I. Mendeleyev Chem Tech Inst, Moscow

"Zhur Obshch Khim" Vol 17, 1947, pp 747-54

Anthrone (I) with strong oleum or $ClSO_3H$ gives ms-anthronesulfonic acid (II). The reaction process is described. The acid was assigned the structure of ms-anthronesulfonic acid on the basis of elementary analysis, presence of dihydroanthrone, anthraquinone, and bianthrone in hydrolysis products, and evolution of some SO_2 during hydrolysis. The change of the yellow color of the mono-Na salt to the greenish color of the di-Na salt also indicates that the latter has an ortho-quinoid structure across the 9,10-ring.

"Mechanism of the Friedel-Crafts Reaction: VI, Reaction of Alkyl Iodides With Benzene," V. V. Korshak, G. S. Kolesnikov, D. I. Mendeleyev Chem Tech Inst, Moscow

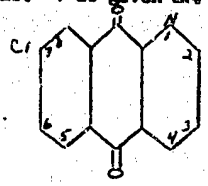
"Zhur Obshch Khim" Vol 17, 1947, pp 1643-4

The reaction of benzene with HI in the presence of $AlCl_3$ was studied with analysis of the HX evolved. In the case of MeI the HCl:HI ratio was 3:1; EtI gave a gas containing 25.95 mol % HCl and 74.15 mol % HI. Similarly, decomposition of EtI with $AlCl_3$ gives 34.17% HCl and 65.83% HI. Mixing HCl in benzene with an equimolecular amount of $AlBr_3$ in benzene followed by heating, gave a gas mixture containing 75.01 mol % HBr and 24.99 mol % HCl. This proves the occurence of the reaction of HCl with $AlBr_3$ in benzene. $AlBr_3$ in warm benzene was slowly treated with an equimolecular amount of HCl or BuCl in benzene, and the gas mixture evolved was analyzed in 4 separate portions; with BuCl as addend, the gas composition varied from 9.31 mol % HCl and 90.69 mol % HBr in the initial stage, to 27.76 mol % HCl and 72.24 mol % HBr in the final determination; when HCl was the addend, the initial gas was 4.5 mol % HCl and 95.5 mol % HBr, going up to 28.36 mol % HCl and 71.64 mol % HBr in the final determination. This indicates that the process has characteristics of an equilibrium reaction; both the HX and RX reactions are very similar.

"Condensation of Quinolinic Acid Anhydride With Chlorobenzene," I. M. Kogan, L. A. Shchukina, D. I. Mendeleyev Chem Tech Inst, Moscow

"Zhur Priklad Khim" Vol 19, 1946, pp 935-30

When quinolinic acid is heated with Ac_2O, it gives the anhydride (I). When I, PhCl, and $AlCl_3$ are heated, treated with HCl, and steam-distilled to remove PhCl, they give 3-para-chlorobenzoyl, icolinic acid-HCl (II). If H_2SO_4 is used instead of HCl, the H_2SO_4 salt is formed. When II is crystallized from H_2O it forms the monohydrate of the free acid, which after drying gives 3-para-chlorobenzoylpicolinic acid (III). III forms a complex Cu salt. The Co, Ni, Fe, Zn, and Pb salts are slightly soluble in H_2O, the Na, Ca, Ba, and Al salts are very soluble. Oxidation of III with alkaline $KMnO_4$ gives para-$ClC_6H_4CO_2H$. III and $SOCl_2$ give a yellow chloride (IV) which with NH_3 gives the amide. IV does not give ring closure with $AlCl_3$ in CS_2 or $PhNO_2$. Heating III with H_2SO_4 gives V. Reduction with Zn dust and NaOH gives a green solution of the corresponding hydroquinone which regenerates V when shaken in air. V is given as:



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